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Animal, Vegetable or Mineral? Characterising shell-lime, maerl-lime and limestone-lime mortar evidence from the Late Norse and Medieval site of Tuquoy, Orkney.

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Abstract

Recent examination of an extensive curated assemblage of mortar samples, recovered from the Late Norse and Medieval site of Tuquoy (Orkney) during excavation in the 1980s, suggested the collection was associated with distinct groups of compositionally contrasting materials related to discrete constructional events. Subsequent petrographic analysis supported this early interpretation and presented evidence for a remarkable series of phase-specific mortars, bound with a range of different biogenic and geogenic lime source materials - including marine shell, coralline algae (*maerl*) and limestone. Wider landscape survey highlighted the broad range of exposed calcareous materials in the coastal and sedimentary environments dominating the Northern Isles of Scotland today, and that many of these different potential lime sources were exploited by craftspeople at different times in the Medieval and later period is now clear.

Given the high significance of the Tuquoy mortar study for our understanding of the development of this culturally important site, and as a prelude to more general publication of the wider archaeological project, a further investigation of selected samples from the mortar assemblage is now being undertaken through a range of geoscientific techniques. This paper presents emerging evidence from a comparative petrographic, SEM-EDS and XRD study designed to further characterise these various mortar materials, and challenge those previous interpretations of contrasting building lime sources. Like most environmental archaeological investigations, this study is essentially concerned with interpreting the depositional histories of surviving materials, but with a particular focus on establishing the distinction between (anthropogenic) kiln relict and (natural) added temper mixtures when both contain biogenic and geogenic clasts.

Introduction

The Late Norse and Medieval settlement site of Tuquoy is located on the south-west coast of Westray, Orkney, in the Northern Isles of Scotland. The upstanding archaeology of Westray from this period is rich and includes the remains of two Medieval and later parish churches, a large late Medieval castle and a Medieval and later multiperiod farmstead, all of which are situated on the more fertile coastal fringes of the island. Indeed, the former parish church of

Cross Kirk at Tuquoy is located within a burial ground which is now situated at the shore edge, just above high tide.

The ruinous remains of Cross Kirk present a bicameral architectural form, wherein a smaller narrower chancel (in this case barrel-vaulted) is situated at the east end of a wider but coeval nave (see figure 1a). This planform is widely distributed at Late Norse sites throughout Scandinavia, Scotland and Ireland, and most of these buildings have been ascribed to the 11th-13th-century on planform alone [1, 2, 3]. The multiphase form of the church at Tuquoy, however, has had increased significance for North Atlantic scholars since Clouston suggested that the extension of the building's nave (to provide room for a larger congregation) provided physical evidence for the emergence of a parish system in the Northern Isles, and for an earlier pre-parochial network of 'ounceland' chapels closely associated with high status farms [4, 5]. One of the classic site-types for this binary Late Norse farm/church configuration in Orkney survives on the very small island of Wyre, where the upstanding remains of a masonry tower and nave-and-chancel chapel are located very close to one another. Moreover, a reference in *Orkneyinga Saga* attributing construction of this tower to Kolbein Hrúga ('Cubbie Roo') before 1150 [6], suggests this may be the earliest masonry castle building surviving in Scotland.

Despite the importance of the Tuquoy church to Orcadian archaeology, however, the form and location of the secular settlement associated with this site remained unknown until the 20th century when 'massive stone walls' began eroding out of a coastal cliff-section approximately 50m west of the upstanding bicameral church [7] (see figure 1b). Moreover, one of these walls was described as 'bearing the same shelly lime render as occurs on high-status medieval buildings such as Cubbie Roo's Castle and The Wirk', and this newly identified site was then subject to a multi-season programme of excavation and environmental sampling in the 1980s [7]. This ultimately revealed the remains of a small multiphase masonry hall and other structures, and excavated deposits from the hall returned a series of radiocarbon dates suggesting occupation in the 12th century [8, <https://canmore.org.uk/c14index/2822>]. Significantly for this paper, the sampling strategy associated with that excavation also included the careful recovery and curation of an extensive assemblage of lime mortar samples.

The mortar assemblage from Tuquoy has now been subject to two programmes of investigation. An initial study was undertaken during a wider research project investigating the Medieval and later masonry mortars of North Atlantic Europe [3], and those initial interpretations are now being re-assessed, challenged and refined as a prelude to publication of the wider archaeology of the Tuquoy site. Both mortar studies will be summarised below.

The Initial Study

Methods 1

The initial study of the Tuquoy mortar materials included: hand sample analysis of the mortar assemblage curated from the hall excavation; field survey of the mortars displayed in the neighbouring church; and thin-section petrographic analysis of selected samples from both sites. Hand sample analysis of all fragments identified as mortar or plaster within the curated assemblage was undertaken by non-intrusive examination of sample surfaces with the unaided eye, a x10 hand lens and a x40 field scope. Subsequent fieldwork in Westray included non-intrusive survey of the upstanding ruined bicameral church at Tuquoy and walkover survey of the adjacent shoreline, with the collection of a variety of loose material samples.

A representative range of mortar samples from both building sites was then selected for thin section preparation, to enable petrographic analysis. These selected fragments were sawn in a variety of planes (relative to wall faces), and dried, before one sawn surface was consolidated with 'epothin' epoxy resin. The consolidated surface was then ground on a horizontal lap to form a flat surface and mounted on a 75 x 26mm slide. Excess material was cut off the mounted sample to allow the material to be lapped, hand polished to a standard 30µm thick, and coverslipped. These prepared thin sections were subject to microscopic examination in plane and polarised light, using a Leica DMLM polarising microscope with image capture by LAS V4.0 software.

XRD analysis was undertaken on two biocarbonate inclusions, hand-picked from selected mortar samples. These materials were ground down by hand in a mortar and pestle to achieve a <50-micron grain size, then scanned in a Bruker D8 Advance x-ray diffractometer using Cu K-alpha radiation filtered to remove the Cu K-alpha 2 peak. The samples were scanned from 2 to 70 degrees 2-theta at a step size of 0.025 degrees and a dwell time of 1.5 seconds per step. The resultant scans were analysed using the Bruker EVA software coupled with the current issue of the ICDD PDF-4 database, with modal analysis carried out using TOPAS 3.0 Reitveld analysis software calibrated and checked against several representative synthetic mineral mixtures.

Hand Sample Analysis

Surface examination of mortar fragments from the excavation assemblage suggested each sample represented a compositionally consistent single-phase material, without layering or stratigraphic horizons. The overall assemblage was clearly comprised of at least three different mortar types, however, and these were characterised according to the following typology:

- **Type A** – A fine, hard and buff-coloured lime mortar included with a high concentration of coralline algae fragments (hereafter *maerl*) which displayed a spectrum of altered colours and textures. These samples generally presented a planar face, with surviving mortar 'tails' on the reverse which had been moulded to the jointing in the face of the underlying rubble wall (see figure 2a).
- **Type B** – A very lime-rich, fine, soft and white-coloured lime mortar containing a low concentration of discoloured *C. edule* shell fragments with distinctively ribbed

morphologies. Also presenting a planar face, with surviving mortar ‘tails’ on the reverse which had been moulded to the jointing in the face of the underlying rubble wall (see figure 2b).

- **Type C** – A fine light-buff lime mortar without visible geogenic or biogenic inclusions but containing a widespread distribution of very fine black grains. These samples presented very thin (6-7mm only) coating profiles and no moulded mortar ‘tails’.

The planar surface displayed by many of the mortar fragments within the curated assemblage suggested the collection was dominated by ‘coating’ fragments, which had been deposited in a plastic state upon and/or within the surface of a masonry wall. Moreover, the smooth texture of the moulded tails and other surfaces suggested that these mortar fragments had been in direct contact with bare rubble stone and were not underlain by earlier mortars in these particular wall face contexts. Most of the mortar samples collected during the 1982-3 excavations were found in stratified deposits and labelled by context, and when mortar was found adhering to a wall this was recorded in the sample labelling (O. Owen pers. com.). Indeed, some of the labels associated with the Type A samples characterised above clearly referred to the ‘hall’ building from which they had been removed, and some labels gave a valuable hint of phasing. Type A sample TWO.04, for instance, was described as “plaster from 1 after removing 2”, with Wall 1 referring to the external face of the hall and Wall 2 part of a substantial workshop immediately adjacent. Although also displaying a mortar tail indicating initial deposition within the surface of a masonry wall, the building from which Type B sample TWO.08 had derived was not immediately clear from its label description of ‘shaped plaster’; the lack of reference to a particular building in this instance probably therefore indicating retrieval from a secondary depositional context. Both Type C sample labels clearly indicated they had been removed from the neighbouring Cross Kirk, however, and the lack of mortar tails in these coating fragments was therefore of some interest.

It is important to note that two of the three mortar types characterised in the above typology displayed biogenic inclusions whose character suggested the lime matrix had been manufactured from biocarbonate lime sources; with Type A samples included with altered skeletal *maerl* fragments with some similarities to the assemblage from Cubbie Roo’s site on Wyre [9], and Type B samples included with altered *C. edule* shell fragments (similar to those from *Eaglais na h’Aoidhe* on Lewis for instance [10]). The lack of visible (potentially kiln relict) carbonates in the Type C samples, therefore, presented a compositional contrast in addition to the morphological differences noted above.

Field Survey

The Tuquoy settlement buildings themselves could not be re-evaluated during this study, as the site had been carefully back-filled after the 1980s excavations, but subsequent fieldwork in Orkney did include a walkover of the adjacent foreshore and a preliminary survey of the neighbouring bicameral church [3].

The ruinous remains of the church at Tuquoy still clearly displayed two distinct phases of construction on the date of survey, although heavy consolidation of the wall faces and some wallhead re-construction effectively precluded comprehensive in-situ characterisation of the structure's mortar archaeology (particularly in the secondary west end of the nave). Various fragmentary patches of mortar indicated the church building had previously been externally coated, however, and more extensive evidence for a very fine mortar coating survived on internal wall faces throughout the primary east end of the building - in both nave and chancel. Three superimposed layers of compositionally contrasting mortars were noted at the east end of the south nave wall, however, and the distinctive brown-coloured and shell-included mortar which underlay this stratigraphic sequence was also visible in the wall core to 280mm deep (within a putlog socket). Acknowledging the limited exposure (resulting from widespread consolidation), limited visibility (the internal coating was obscured by organic growth whilst the external coating was very fragmentary), and general lack of continuity between core and coating (resulting from re-pointing), the evidence was regarded as sufficient to suggest these mortars were constructional materials and a single loose sample of the brown-coloured shell-included mortar was collected from within the core of the south nave wall (adjacent to the putlog). This sample was characterised in hand sample as follows:

- **Type D¹** – Sample TWO.09 is hard brown-coloured lime mortar included with a fine mixture of lustrous shell fragments (grading to 2-3mm), but without any clearly identifiable relict limekiln evidence.

Petrographic Analysis 1

Petrographic analysis was undertaken on mounted thin sections prepared from nine selected mortar samples. This included at least one section from each putative mortar type but, with a particular concern to further investigate the potential use of *maerl* as a lime source, the sub-assemblage was dominated by samples previously characterised as Type A materials.

The main characteristics of each sample Type in polarised light are summarised below:

- **Type A** samples (TWO.01, TWO.02, TWO.04, TWO.06 and TWO.07) generally display a bimodal matrix-supported texture², with a coarse fraction containing *maerl* and marine shell fragments grading to 8mm diameter (see figure 3a). These biogenic clasts present a spectrum of altered textures, including increased micritisation, loss of internal microstructure, loss of grain boundary coherence, and increased optical continuity with the supporting carbonate matrix, such that any distinction between the bioclast and matrix is often ambiguous. The carbonate matrix is generally cryptocrystalline to microcrystalline with more dense areas often resolving to concentric and/or cellular relict biogenic forms consistent with skeletal *maerl*. A low concentration of highly altered quartz-rich geogenic clasts are also evident in these sections, with clasts displaying distinct grain boundaries but also isotropic properties, high vesicle concentrations and some spiniflex textures. Possible relict fuel inclusions are very rare in these thin sections, but some opaque and irregular possible relict peat

evidence was noted. These mortars have generally been tempered with a well-sorted submillimetric mixture of subangular monocrystalline quartz and marine shell (grading to 0.25mm) with rare feldspar and larger subrounded quartzose grains included.

- **Type B** sample (TWO.08) is also bimodal, but with an extraordinary high volume of very fine ('cloudy') cryptocrystalline carbonate matrix supporting a very low concentration of poorly-sorted clasts grading to 5mm (see figure 3b). This coarse fraction is dominated by a mixture of altered marine shell fragments (including *C. edule*) with a lower concentration of altered geogenic clasts with some spinaflex textures. The section includes a moderate concentration of opaque probable peat fuel relicts, and a lithic/shell temper mixture dominated by fine (submillimetric) marine shell fragments.
- **Type C** sample (TWO.03) is a well-sorted and very fine mortar material which displays a remarkably high concentration of altered quartz-included geogenic clasts forming an interconnected network of 'globular', vesicular and isotropic reaction products (with indistinct grain boundaries) which are closely associated with widespread evidence for very fine altered biogenic clasts (see figure 3c). The mortar displays a low concentration of fine, irregular and opaque probable peat fuel relicts and is tempered with a fine mixture of monocrystalline quartz and marine shell.
- **Type D** mortar sample (TWO.09) presented a matrix-supported poorly-sorted mixture of biogenic and geogenic grains grading to 3mm (see figure 3d). Identifying relict lime-source carbonates within these sections was challenging, but two large rounded areas of amorphous carbonate and localised scatters of fine quartz (interpreted as intracrysts released from a quartz-included lime-source 'protolith') were regarded as probable evidence for a geogenic lime-source which was tentatively identified as a micritic limestone/mudstone. These sections did also contain a very low concentration of micritic and fractured *maerl* fragments, although these did not appear to have a textural relationship with the supporting carbonate matrix, whilst the fine shell within the temper mixture appeared largely unaltered. A low concentration of fine probable relict peat fuel inclusions was also noted.

XRD Analysis 1

To further characterise the biocarbonate inclusions within the mortars of the curated assemblage, multiple *maerl* (TQ2) and *C. edule* shell (TQ8) inclusions were picked out from Type A sample TWO.02 and Type B sample TWO.08 and submitted for XRD analysis. These analyses returned similar ratios of different carbonate polymorphs - dominated by high calcium calcite (59-71%), with significant magnesium calcite (24-32%) and almost trace (1.2-1.3%) aragonite fractions. Unlike TQ8, however, *maerl* sample TQ2 also returned very minor levels of periclase (1.3%) (see table 1 below).

Summary of the Initial Study

The initial study of mortar samples retrieved from the excavated settlement and upstanding church at Tuquoy highlighted evidence suggesting the combined assemblage was comprised of four compositionally contrasting mortar materials. Within this typology, mortar Types A and B (from the settlement excavation) included high concentrations of altered biogenic (*maerl* and marine shell) grains, with lower concentrations of altered quartz-rich geogenic clasts. The altered micritic/cryptocrystalline texture of these biogenic clasts, and their intimate textural and optical relationship with their supporting (lime mortar) carbonate matrices, was interpreted as evidence that these Type A and Type B materials were *maerl*-lime and shell-lime mortars respectively. Mortar Types C and D (from the upstanding church) were both more tentatively interpreted as geogenic-lime mortars, largely on the basis of various altered quartz-rich clasts which in Type C were in remarkably high concentration.

In-situ evidence suggesting that *maerl* had been exploited as a building lime-source at Cubbie Roo's castle and chapel on Wyre, had already been identified when the curated assemblage from the Tuquoy excavation became available for examination [9]. The *maerl*-lime mortar evidence reported during this initial study of the Tuquoy hall mortars, therefore, is consistent with Lamb's observation that these structures were coated with similar materials [7] (see above) and parallels the broad contemporaneity of these various Orcadian buildings. The compositional contrasts upon which the Tuquoy mortar typology was based, however, suggest that this Westray site is associated with at least three separate lime-bonded constructional events, and that (unlike the Wyre buildings) the church and hall at Tuquoy had been constructed at different times [3]. Since accepting this interpretation would have significant implications for our understanding of the development of the site, further geoscientific study of these complex materials was commissioned to reassess those interpretations.

A Materials Reassessment

Methods 2

A reassessment of the Tuquoy mortar evidence was undertaken on samples selected from the initial study, and included SEM-EDS, further XRD, and further petrography analysis. For the SEM-EDS analysis, polished thin sections of Type A sample TWO.04, Type B sample TWO.08, and Type D sample TWO.09 were prepared and carbon-coated. These slides were analysed using a Hitachi S4100 Field Emission Scanning Electron Microscope (FESEM) using Backscattered Electron Imaging at 20kV accelerating voltage, with chemical analysis performed using an Oxford Instruments Energy Dispersive Spectrometer (EDS). The second round of XRD employed the same methods as the initial study, but examined various different materials including: fine <63µm fractions of three selected mortar fragments which had been crushed in a mechanical jaw crusher then sieved to remove the coarser fractions; further selected mortar inclusions picked-out from the surface of mortar fragments using a sharp point; and loose fragments of detrital materials collected during walkover of the Tuquoy shoreline. These latter materials included a small sample of aeolianite, from which another

30µm thin section was also prepared. Finally, armed with evidence reported from the SEM-EDS and XRD analyses, a further round of petrographic analysis was undertaken at the University of Stirling, using an Olympus BX51 polarising microscope and analySIS pro imaging software.

SEM-EDS Analysis

Samples from each of the three main constructional mortar types (so excluding Type C coating samples) were subject to SEM-EDS analysis, to further examine the relationships between various (biogenic and geogenic) inclusions and the supporting carbonate matrices. With reference to figures 4a-e, the results of this study and a summary of interpretations are presented below:

- **Type A** sample TWO.04 presents a matrix-supported mixture of high Ca *maerl* and shell fragments, whilst a porous fragment of material containing both Ca and P suggests some bone fragments may also be present. Notably, the general carbonate matrix also presents a high Ca composition, often with a small P and S signature but with no appreciable Mg content. The section contains some sedimentary sandstone, but elemental mapping suggests this lithology has no Ca-bearing phase.
- **Type B** (sample TWO.08) presents a nearly pure high-Ca matrix, supporting shell fragments with a similar chemistry and porous texture. This section also contains some remnant bone fragments (here with some altered halos) and widespread evidence for highly altered subangular fragments of quartz-included slag, with well-developed (spherical) vesicles and some spiniflex textures.
- **Type D** (sample TWO.09) presents a more heterogeneous calcareous matrix with an appreciable Si, Mg and Al content consistent with a fine included aggregate or possibly a hydraulic phase. A quartz grain with a halo of Si-Ca, Al and Mg, and another quartz-included Ca-Si slag with a diffuse grain boundary, was also noted. The section also contains large subangular siltstone fragments with a well-sorted and calcareous composition (characterised here as a calc-arenite), as well as high calcium shell fragments.

In summary, SEM-EDS analysis indicates that the mortar matrix in both the Type A (TWO.04) and Type B (TWO.08) samples was composed of a high Ca-carbonate with only minor evidence for other elements such as Mg, S and Fe, and this is indicative of a carbonate source comparable to the biogenic clasts which are widely distributed throughout these sections. This interpretation is consistent with the pseudomorphic features visible in calcined lime inclusions of similar dimensions to neighbouring biogenic clasts, although it was not initially clear how this evidence relates to the abundant Ca-Si slag fragments in Type B sample TWO.08 since these clearly suggest the raw material contained a high proportion of silicates (see summary and discussion below).

In contrast to these Type A and B mortars, the supporting carbonate matrix in Type D sample TWO.09 presented a consistently higher Si and Al content. The abundant grains of calc-arenite in this section were not noted in previously considered sections and might be considered as a possible lime-source material, although, except for the apparently rare grains of reacted quartz, there is a lack of obvious clinker phases in the section.

XRD Analysis 2

Further investigations of selected materials from the Tuquoy assemblage was undertaken using XRD, with a particular concern to examine the polymorphic mineralogy of various calcareous materials, including mortar inclusions, mortar matrices and some environmental materials. The background to this study was provided by recent experimental work which had demonstrated that Orcadian *maerl* gravels are dominated by high-magnesium calcite, which undergoes a predictable series of mineral phase changes with increasing temperature, whilst *C. edule* shells undergo parallel changes from aragonite to (low-Mg) calcite [3]. The polymorphic profile of these biogenic inclusions from historic mortars can then be used to characterise thermal histories, as had been previously done during the Cubbie Roo's Castle study [3]. Essentially, this same approach was being adopted to establish whether selected inclusions within the Tuquoy Type A mortar had undergone heating (so were a probable lime source) by comparison with (presumably unheated) detrital samples from the adjacent foreshore.

Including the investigations undertaken during the initial study (see above), XRD analysis was undertaken on the following materials:

(i) fine sieved fractions (<63µm) from jaw-crushed fragments of Type A (TWO.06), Type B (TWO.08) and Type D (TWO.09) mortar samples;

(ii) *maerl* inclusions (TQ2 & TQ4) removed by hand from the surface of two different Type A mortars (TWO.02 & TWO.04);

(iii) *C. edule* shell (TQ8) inclusions removed by hand from the surface of a Type B mortar (TWO.08);

(iv) a *maerl* inclusion (TQ3) collected from the foreshore close to the Tuquoy settlement site; and

(v) a loose fragment of aeolianite (TQ10) collected from beneath an exposure close to the Tuquoy settlement.

Table 1. Sample compositions by XRD analysis.

Sample	Mg-Calcite	Calcite	Periclase	Dolomite	Aragonite	Trace + Silicates
Maerl						
TQ2	24	71	1.3	-	1.3	2.4
TQ3	11	86	-	-	-	3
TQ4	-	94	-	-	-	6 ¹

Shell						
TQ8	32	59	-	-	1.2	7.8
Aeolianite						
TQ10	6	56	-	4.1	8.7	25.2 ²
Mortar						
TWO.06	-	76	-	-	4	20 ³
TWO.08	-	96	-	-	-	4 ⁴
TWO.09	1.5	78	-	-	2.3	18.2 ⁵

All figures are percentages of total composition; all figures less than 1% are regarded as 'trace'; all carbonate polymorph figures are rounded up to nearest integer; Trace + Silicates: 6¹ in TQ 4 includes 3% quartz & 1% microcline; 25.2² includes 12% albite, 9.7% microcline & 3.4 muscovite; 20³ in TWO.06 includes 7.4% quartz, 5.5% mullite & 3.5% microcline; 4⁴ in TWO.08 includes 1% quartz & 1% albite; 18.2⁵ in TWO.09 includes 10% quartz, 4.3% albite, 1% microcline & 1% muscovite.

The results indicate that the polymorphic profile of all three mortar fine fractions is dominated by (low-Mg) calcite, although small metastable polymorph concentrations are evident in Type D sample TWO.09 (see table 1). The relative concentration of silicates in the fine fraction data from all three mortars appears to correlate with the relative concentrations of fine temper noted during thin section analysis, however, and this is supported by the relatively high concentrations of quartz within Type A sample TWO.06 and Type D sample TWO.09. The most notable detail within this data, therefore, is the continued evidence for very low temper (and low quartz) concentrations in Type B mortar sample TWO.08.

All biocarbonate materials, including the mortar inclusions and the detrital foreshore *maerl* sample TQ3, are dominated by (low-Mg, high-Ca) calcite. The complete lack of high-Mg calcite in mortar inclusion TQ4, and the relatively low fraction of this same polymorph in detrital sample TQ3 are notable.

Petrographic Analysis 2 & Reassessment Summary

With reference to the emerging SEM-EDS and XRD reports, a petrographic re-evaluation of the Tuquoy thin section assemblage (with the addition of a recently prepared thin section of aeolianite sample TQ10) was undertaken to inform final interpretations. This re-assessment also prompted a further round of analysis on the SEM-EDS geochemical data as the reflexive cycles of this investigation continued.

The very close optical and textural relationship between included bioclasts and supporting carbonate matrices in Type A and Type B mortars (noted in the initial study) is striking. The evidence for altered shell material in Type A mortars is perhaps more salient than previously reported, but the heterogeneity in the temper profiles of these Type A sections² lends further support for the *maerl*-lime and shell-lime interpretations of these Type A and B materials. SEM-EDS analysis indicates that these (biogenic and anthropogenic) Type A and B materials also have very similar high-Ca compositions with no appreciable Si content (see figure 4a & 4b). This high-Ca evidence is consistent with the XRD data from *maerl* inclusion TQ4, removed from this same SEM-EDS analysed mortar sample (TWO.04), and with the fine fraction from

Type A mortar sample TWO.06. There is a lack of evidence within the current XRD data to suggest a correlation between *maerl* mineralogy and depositional context in the Tuquoy assemblage, as the foreshore *maerl* clast TQ3 displays much less high-Mg calcite than expected from previous analyses of Orcadian materials, and even presented a lower concentration of this polymorph than the *maerl* mortar inclusion TQ2 from Type A sample TWO.02. The low levels of high-Mg calcite in the mortar inclusions of TQ2 and TQ4, and the low level of aragonite in the *C. edule* mortar inclusions of TQ8, however, are both consistent with the polymorphic inversion associated with heated kiln relicts of these types [3]. The lack of aragonite and high-Mg calcite in the fine fraction from sample TWO.08 is also consistent with the high-Ca chemistry of the matrix of this same mortar sample, suggested by the EDS data, and with the XRD results returned by shell-lime mortar fine fractions from other sites [3]. The mineralogy and geochemistry of these materials in a naturally deposited environmental context therefore clearly requires more work (only a single *maerl* fragment was analysed here), but the textural, optical and geochemical characteristics of these bioclastic materials in the mortars themselves presents convincing positive evidence for a biogenic lime-source in these Type A and Type B mortar fragments.

Whilst there is an apparent lack of part-calcined geogenic calcareous lime source materials in all the Tuquoy mortar samples examined in these studies (such as those described in [11] for example), these mortars are often clearly associated with evidence for altered geogenic clasts of some sort, and these present a range of forms. Indeed, petrographic analysis suggests that at least some of the altered geogenic clasts visible in the Type A mortar thin sections have been formed from very rounded (detrital) sandstone clasts which display a spectrum of altered textures - including irregular clast boundaries and isotropic phases but no very significant calcareous component (see for example figures 2a and 3a). These clasts often appear to be at an early stage of alteration, with vesicular and isotropic glassy materials sometimes limited to narrow areas and 'blisters' around clast rims. Importantly, the apparent lack of a significant calcareous component within these altered rounded clasts is consistent with the EDS data from a similarly red-coloured ferruginous grain in TWO.04, wherein normalised spectra analysis indicates a CaO fraction of 3.92-6.13% only (see figure 4c). More highly altered geogenic textures are visible in Type B sample TWO.08, in broken angular fragments of well-developed previously liquid phase glassy materials with abundant well-formed crystals and some spinaflex textures (see figure 3b), and EDS analysis indicates these 'slags' contain a CaO fraction ranging from 3.94-19.62% (see figure 4d & 4e). The low calcareous fraction within these altered silica-rich geogenic clasts suggests they are incidental relicts of the limekiln charge, and not lime-source materials.

Type C sample TWO.03 was not subject to SEM or XRD analysis, but a better interpretation of this material has been informed by comparative microscopic analysis of an aeolianite sample collected during walkover of the Tuquoy foreshore. TWO.03 is a fine well-sorted mortar of very similar texture to the aeolianite thin section TQ10 but contains an extraordinarily high concentration of slaggy clasts which are not apparent within this latter naturally deposited

material. Moreover, unlike in thin section TWO.04 (Type A) and TWO.08 (Type B), the slags in thin section TWO.03 are not fragmented but appear to have derived from very fine geogenic protoclats which have a close association with neighbouring fine altered bioclats (see figure 3c). It is this material which accounts for the distinctive fine black specks seen in the Type C hand samples during the initial study (and not noted in other mortars in the assemblage), and on this basis an aeolianite lime-source is suspected.

The initial study suggested that Type D sample TWO.09 had been manufactured from a geogenic lime source on the basis of both negative and positive evidence. The marine shell fraction within TWO.09 displays an unaltered range of colours and variously lustrous textures in hand sample, and this is consistent with the high birefringence, surviving microstructural integrity and lack of relationship with the supporting carbonate matrix associated with these grains in thin section. Overall, the evidence suggests that this mortar was not manufactured from a biogenic lime source. The initial study identified various subangular clasts of a fine-grained calc-sediment as a probable lime-source, and the SEM-EDS evidence identified a Ca-bearing lithology (characterised as a calc-arenite in sample TWO.09) as a possible lime-source also. It has not been possible to identify clearly part-calcined lime-source relicts in this reassessment of this material, but the identification of localised scatters of fine quartz within TWO.09 in the initial study, and their characterisation as intraclasts released during calcination of the geogenic parent lime-source, is consistent with the evidence for reacted quartz grains reported during the SEM work. The identification of Si, Al and Mg in the cryptocrystalline carbonate matrix of this material also clearly supports a geogenic lime provenance, with some Mg also evident in the XRD data from the TWO.09 fine fraction. This evidence for a Si, Al and Mg bearing carbonate matrix in TWO.09 presents a clear contrast with the biogenic mortar Types A and B described above, as does the lack of evidence for large isotropic/vesicular slags which were so clearly evident in the biogenic-lime materials.

A comparative summary of these various compositional criteria for mortar Types A, B, C and D, is presented in Table 2 below:

Table 2. Summary of Mortar Compositions by Type

Mortar Type	Grain Distribution	Carbonate Relicts	Slag Relicts	Fuel Relicts	Matrix Composition	Added Temper
A	Bimodal	Maerl/Shell	Poorly developed	Peat	High-Ca	High concn. Sub-mm shell & quartz
B	Bimodal	Shell/Maerl	Well-developed	Peat	High Ca	Low concn. Sub-mm shell & quartz
C	Well-sorted	Aeolianite	Complex	Peat	Unknown	Low concn. Sub-mm shell & quartz
D	Poorly-sorted	Limestone	Minimal	Peat	Ca with Si, Al & Mg.	Mod. concn. shell & quartzose

Conclusion

A reflexive programme of hand sample examination, petrographic thin section analysis, SEM-EDS analysis and XRD analysis of a mortar assemblage from Tuquoy in Westray (Orkney) has presented a consistent suite of evidence suggesting that the assemblage curated from the excavated settlement is dominated by biogenic-lime mortars (Type A and B), manufactured from *maerl* and marine shells, whilst the materials collected from the upstanding church are geogenic-lime mortars (Type C and D). Compositional variations and contrasts within this binary interpretation suggest that four different mortar types (A-D) are represented in the overall assemblage.

Discussion

Evidence for phase-specific lime-source contrasts in single multiphase sites is widespread across the north and west of Scotland and, at least in part, the broad range of different biogenic and geogenic lime-source materials used in the Northern Isles throughout the Medieval and later periods reflects the wide range of calcareous resources available in this coastal sedimentary environment [3]. This wide distribution of calcareous materials, however, also often results historic mortars in which both kiln-relicts and added-temperers are composed of various mixtures of altered geogenic and biogenic materials with complex depositional histories, and this can make lime provenance interpretations challenging. In the case of the Tuquoy assemblage, this complexity is heightened by the high geogenic/biogenic variability in foreshore compositions noted during walkover survey, and the close proximity of aeolianite outcrops which are also composed of carbonate cemented biogenic and geogenic grains. Indeed, detached and eroded fragments of aeolianite also currently contribute to the detrital materials covering the Tuquoy foreshore, adding even further cycles of complexity to the depositional profile of this environment.

The Tuquoy mortar materials are essentially composed of complex mixtures of closely associated biogenic, geogenic and anthropogenic materials, with altered characteristics resulting from primary, secondary and often tertiary depositional processes. A robust interpretation of these complex materials has required a reflexive interdisciplinary approach to materials analysis, with a particular focus on examining the relationships between these various altered inclusions and the supporting carbonate matrix. All four mortar types characterised in this study display evidence for altered biogenic and geogenic materials, and these appear closely associated in mortar types A, B and C. Elsewhere, an apparent correlation between altered biocarbonate and non-calcareous geogenic clasts has now been reported in several shell-lime mortars across North Atlantic Europe [3], suggesting that both materials are relicts of the limekiln charge [13]. This re-assessment of the Tuquoy assemblage has effectively demonstrated the potential for SEM-EDS analysis to usefully inform that discourse, through the comparative analysis of binder, lime-source and slag geochemistry.

The emergence of slags in limestone-lime mortars is often interpreted as the result of ‘over-burning’ or impurities in the limestone lime source, but the vitreous materials in the Tuquoy assemblage are not consistent with this interpretation. A detailed study of slag inclusions

within limestone-lime mortars manufactured at Charlestown (Fife, Scotland), for example, demonstrated that these materials retained a mineral composition dominated by Ca-rich silicate minerals, with a CaO fraction of between 40.8 and 56.2% [12]. The slag inclusions in Type A and B mortars from the Tuquoy assemblage, in contrast, often appear to have formed from silicate-rich sandstone materials with a range of low calcareous compositions. There is no convincing petrographic evidence for a textural relationship between these altered geogenic clasts and the supporting carbonate matrix in either mortar type, and the lack of evidence for Si phases in the SEM-EDS data from these mortar matrices also appears inconsistent with a high-silicate lime-source material (see figures 4d & 4e). These glassy vesicular fragments require further work, but do not appear to be the product of a geogenic lime source material which has differentiated into calc-silicate slag fragments and Ca-rich binder. As above, it appears more likely that these belong to a class of vitreous fragments noted within historic and experimental materials elsewhere across Scotland, where they have been interpreted as evidence for the incidental inclusion of foreshore gravels within a limekiln charge dominated by a biogenic lime-source [13, 3]. Ultimately, therefore, the altered geogenic evidence in these materials actually lends further support to a biogenic lime source interpretation for mortar Types A and B.

The compositional contrasts observed across the combined Tuquoy mortar assemblage suggest that each of the mortar types characterised in the above investigation is associated with a separate constructional event, and on this basis the initial study suggested that (unlike at the Wyre site of Cubbie Roo's Castle) the hall and church at Tuquoy had been constructed at different times [9, 3]. The recent reassessment of these mortars presented above has supported the materials interpretations which emerged from that initial study, and this effectively turns attention back to the contexts from which these samples were retrieved. With further evidence for the development of these remarkable buildings emerging from other research undertaken for the wider Tuquoy project, the contexts from which these mortar samples were retrieved will be reconsidered in the forthcoming monograph publication [14].

Further Work

The XRD results from the *maerl* analysis highlight a requirement for much more representative sampling and analysis of environmental materials, and at Tuquoy this comparative approach should be extended to include petrographic analysis of locally available temper sands. The interpretation that mortar Type C sample TWO.03 has been manufactured from aeolianite should be followed up by a comparative study of the shell evidence, since initial examination suggests neither the aeolianite nor TWO.03 contain evidence for larger shells such as *C. edule*. Further analysis should also re-consider the bone evidence apparent in Type A and B mortars, since some evidence for P in the carbonate matrix was noted. A further mortar survey of the chapel would be very useful and, although currently precluded by extensive consolidation, future work should aim to retrieve multiple mortar samples from fixed primary constructional core contexts.

Endnotes

1 – Although clearly recognised as contrasting materials, the church samples TWO.03 and TWO.09 were both labelled Type C in the initial study. Mortar Type D has been introduced here for greater clarity.

2 – TWO.06 and TWO.07 are less clearly bimodal.

References

1 Fleming A, Woolf A (1992) Cille Donnain: a Late Norse church in South Uist. *PSAS* 122: 329-350.

2 Thacker M (2015) Cille Donnain Revisited: negotiating with lime across Atlantic Scotland from the 12th-century. *Journal of the North Atlantic* 9: 45-66.

3 Thacker M (2016) Constructing Lordship in North Atlantic Europe: the archaeology of masonry mortars in the Medieval and later buildings of the Scottish North Atlantic. 3 vols. Unpublished PhD thesis, University of Edinburgh.

4 Clouston J (1932) A history of Orkney. MacKintosh, Kirkwall.

5 Gibbon S (2007) Medieval parish formation in Orkney. In B Ballin Smith, S Taylor, G Williams (eds.) *West over Sea. Studies in Scandinavian sea-borne expansion and settlement before 1300*. Brill, Leiden. Pp. 235-250.

6 Pálsson H, Edwards P (1978) *Orkneyinga Saga. The History of the Earls of Orkney*. Penguin. Middlesex. P. 155.

7 Lamb R (1993) Tuquoy: The Norse Magnate Farmstead. In C Morris (ed.) *Congress Diary*. pp. 1-101. In C Batey, J Jesch & C Morris (eds.) *The Viking Age in Caithness, Orkney & the North Atlantic*. Edinburgh University Press, Edinburgh. P. 82.

8 Owen O (1993) Tuquoy, Westray, Orkney: a challenge for the future? In C Batey, J Jesch, C Morris (eds.). *The Viking Age in Caithness, Orkney & the North Atlantic*. Edinburgh. Pp. 318-339.

9 Thacker M (2013) The Late Norse ‘coral’ or maerl-limes of Orkney: an on-site mortar archaeology of Cubbie Roo’s castle and chapel. *Proceedings of the 3rd Historic Mortars Conference, Glasgow 2013*. Rilem, Glasgow.

10 Thacker M (2011) The lime and shell-lime mortars of the Western Isles. Unpublished MA thesis, University of York.

11 Hughes J, Cuthbert S (2000) The petrography and microstructure of medieval lime mortars from the west of Scotland: implications for the formulation of repair and replacement mortars. *Materials & Structures* 33: 594-600.

12 Leslie A, Hughes J (2004) High-temperature slag formation in historic Scottish mortars: evidence for production dynamics in 18th-19th century lime production from Charlestown. *Materials Characterisation* 53: 181-186.

13 Thacker M (2013) Making lime at the edge of the world. Proceedings of the 3rd Historic Mortars Conference, Glasgow 2013. Rilem, Glasgow.

14 Owen O (in prep.). Tuquoy: Investigations at a Norse manorial farm in Westray, Orkney.

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Figures



Figure 1a (above) – Crosskirk Tuquoy from the south-east. Note the putlog socket between nave doorway and window. Scale 500mm; photograph Mark Thacker.



Figure 1b (above) – 50m east of the upstanding church. The eroding cliff-section containing Tuquoy hall and settlement from the south. Scale 500mm; photograph Mark Thacker.



Figure 2a



Figure 2b

Figure 2a (above) – Thick section mortar Type A x 2. Note: thin mortar 'tails' (with coherent rounded ends which may have abutted underlying constructional clay mortar in the masonry joint) and the more planar face of the former mortar surface. Field of view approx. 120mm; Photograph M. Thacker;

Figure 2b (above) Thick section mortar Type B. Note very wide mortar tail suggesting much coarser underlying rubble wall; planar face of previous coating surface; high concentration of discoloured bioclasts including probable *maerl* and shell in this particular section. Field of view approx. 100mm; Photograph M. Thacker.

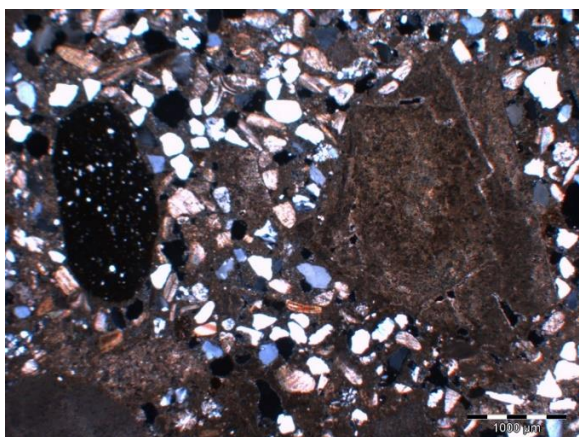


Figure 3a (above)

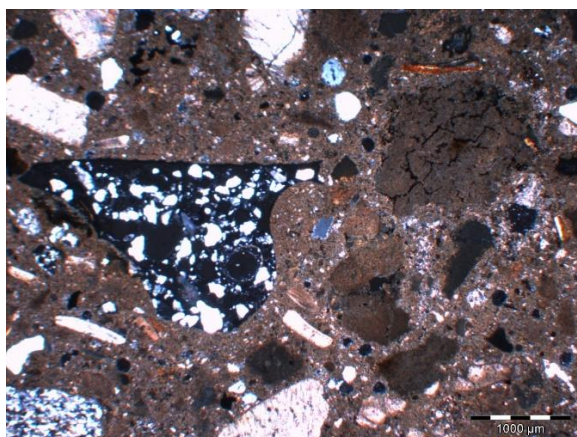


Figure 3b (above)

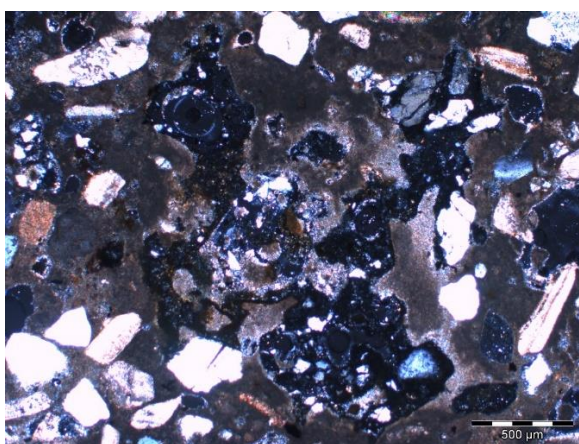


Figure 3c (above)

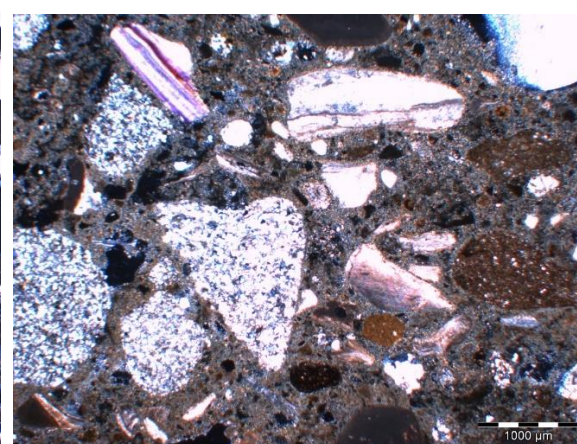


Figure 3d (above)

Figure 3a (above) – Subrounded kiln relict *maerl* fragment close to optical continuity with general mortar matrix in mortar Type A sample TWO.02, with adjacent rounded and isotropic sandstone clast. XPL; Scale bar 1000µm; photomicrograph M Thacker.

Figure 3b (above) – Well developed broken slag fragment and highly altered shell fragments in bimodal Type B mortar sample TWO.08. XPL; 1000µm; photomicrograph M Thacker.

Figure 3c (above) – Delicate network of calc-silica reaction products and altered biogenic grains in well-sorted mortar Type C sample TWO.03. XPL; Scale bar 500µm; photomicrograph M Thacker.

Figure 3d (above) – Poorly-sorted mixture of lustrous marine shell and calc-arenite in mortar Type D sample TWO.09. XPL; Scale bar 1000µm; photomicrograph M Thacker.

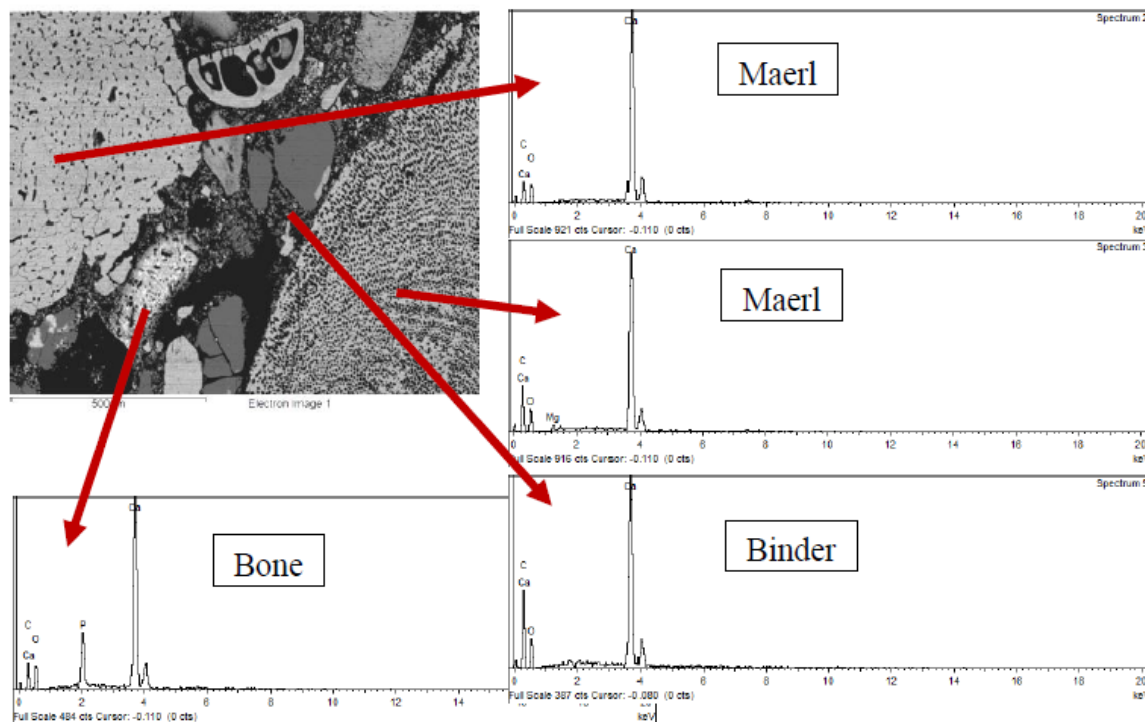


Figure 4a (above) – SEM image of an area of Type A mortar sample TWO.04, with associated EDS spectra for two *maerl* fragments of varying textures, one probable bone fragment and the supporting matrix. Scale bar 500μm; SEM Image John Hughes.

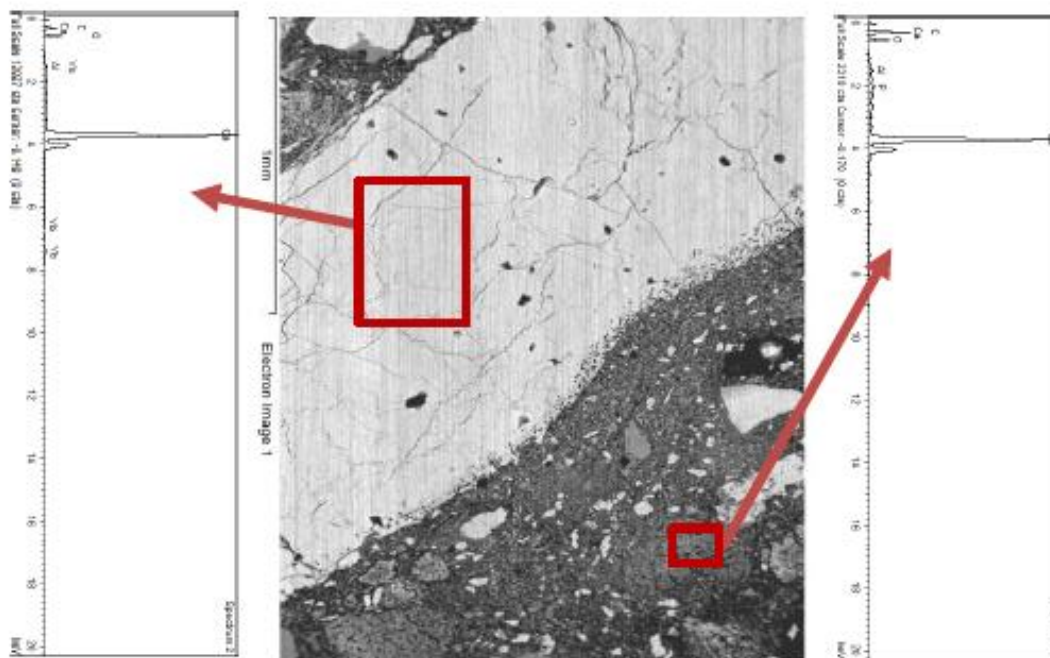
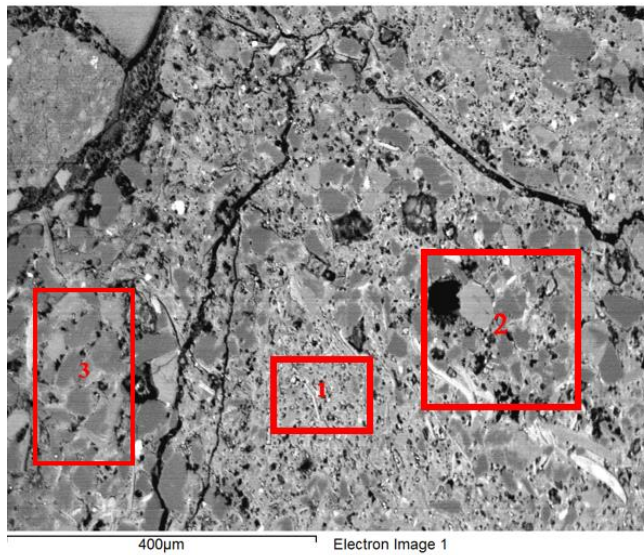
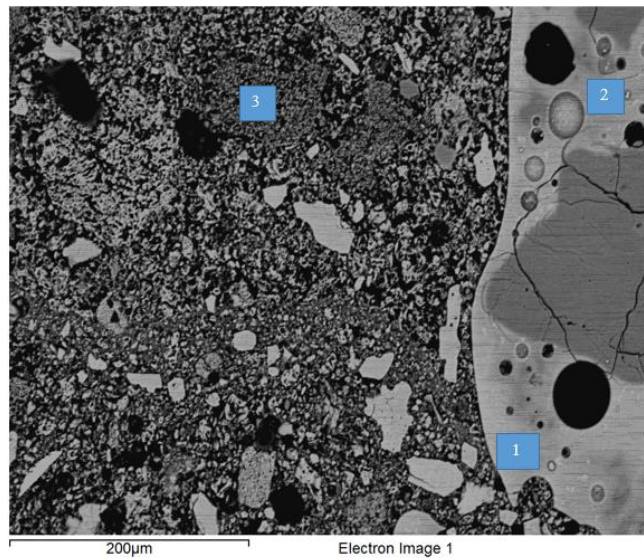


Figure 4b (above) – SEM image of an area of Type B mortar sample TWO.08, with associated EDS spectra for an area of shell and supporting carbonate matrix. Scale bar 1mm; SEM image John Hughes



Spectrum Area	Compd%		
	1	2	3
Na ₂ O	3.67	3.49	3.9
MgO	5.57	5.58	1.85
Al ₂ O ₃	14.42	15.25	13.08
SiO ₂	46.53	46.04	64.48
P ₂ O ₅	0.64	1.03	-0.02
SO ₃	0.27	0.26	0.37
K ₂ O	6.76	6.78	5.29
CaO	5.51	6.13	3.92
TiO ₂	2.74	2.18	0.79
FeO	13.88	13.25	6.34
Normalised	99.99	99.99	100

Figure 4c (above) – SEM image of large rounded sedimentary grain in Type A mortar sample TWO.04. Numbered areas relate to EDS analyses in adjacent table. Scale bar 400µm; SEM image John Hughes.



Spectrum Area	Compd%		
	Slag 1	Slag 2	Binder 3
Na ₂ O	6.91	6.77	0.24
MgO	2.39	1.59	0.24
Al ₂ O ₃	9.86	12.65	1.04
SiO ₂	57.54	63.51	0.7
P ₂ O ₅	2.18	1.58	2.29
SO ₃	0.04	0.17	0.91
K ₂ O	4.74	5.55	0.02
CaO	7.13	3.94	94.56
TiO ₂	2.02	-	-
FeO	7.19	4.24	-
Normalised to	100	100	100

Figure 4d (above) – SEM image of Type B mortar sample TWO.08. Numbered areas relate to EDS analyses in adjacent table. Scale bar 200µm; SEM image John Hughes.

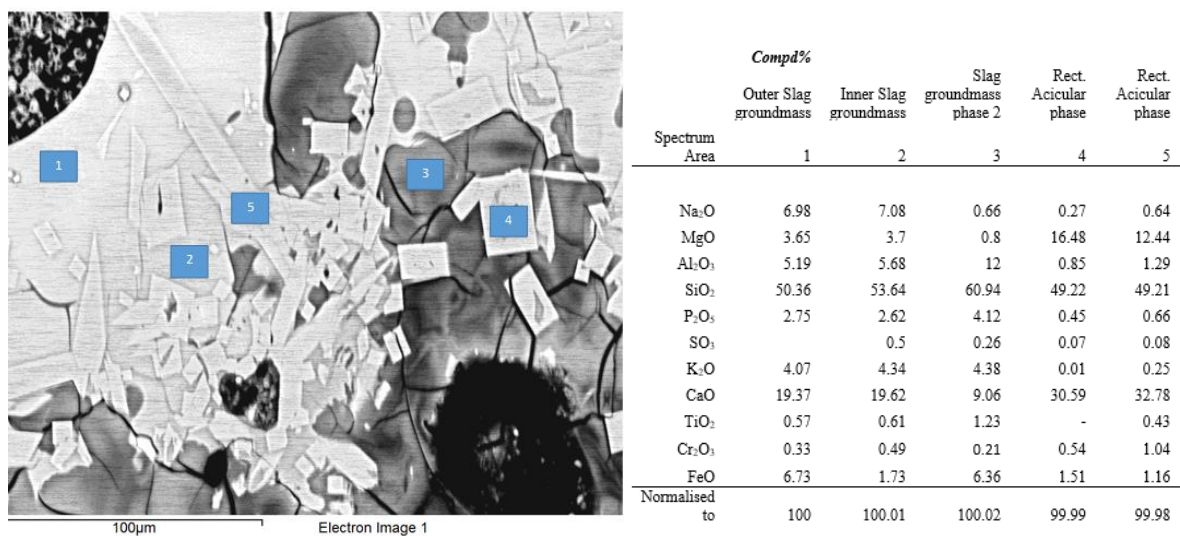


Figure 4e (above) – SEM image of part of a slag grain in Type B mortar sample TWO.08. Numbered areas relate to EDS analyses in adjacent table. Scale bar 100µm; SEM image John Hughes.